

Pre-Hispanic Au-Pt Alloys Experimental Simulation Using Solid State Diffusion

M.E. Noguez^{*1}, G. Salas², J. Ramírez³

Departamento de Ingeniería Metalúrgica, Universidad Nacional Autónoma de México

Facultad de Química, Edificio D, Ciudad Universitaria, C.P. 04510, México, D.F. México

^{*1}nogueza@unam.mx; ²salasb@unam.mx; ³jgrv@unam.mx

Abstract

Au-Pt alloys made in South America from 300 BC-400 AD by the Pre-hispanic inhabitants from Tumaco-La Tolita region (south Colombia-north Ecuador) exemplify an engineering way of solving troubles when making alloys using high melting point raw materials as Pt (1768°C). In this paper metallurgical work has been done simulating experimentally the fabrication of several Au-Pt Pre-hispanic alloys using only solid state processing at 950°C: 88%Au - 12% Pt, Pt Plated Au alloy and Pt - Pt sheets. The employed manufacturing process is presented as a technical possibility compared with the one normally accepted and experimentally experienced by the authors of this work, consisting of agglomeration of Pt particles by melted Au, which has been reported elsewhere. The viability of using pure solid state processes in Pre-hispanic times is evident according to what it is known now about solid state diffusion processes and because of the experiences presented.

Keywords

Pre-hispanic Alloys; Gold-platinum Alloys; Solid State Diffusion; Diffusion Alloying

Introduction

Modern Archaeo-metallurgical methods consider the fabrication of alloy and object replicas as an important step towards the study of the alloys and the involved manufacturing process. Reproducing the alloys using several metallurgical alternatives, based on the studies made to the archaeological alloys, leads to the most probable metallurgical model of fabrication [Escobar and Bustamante, 2011].

Archaeologists have reported the Au-Pt alloys findings in South America: north Ecuador-south Colombia in Tumaco-La Tolita region and are dated in 300 BC – 400 AD, Figure 1. From D. Scott and W. Bray [1994] Au-Pt objects archaeological classification, the diversity of the Au-Pt alloys is as follows: 1) native Pt; 2) “sintered” Au-Pt¹ from 0% to 70% Au and 3) Pt

plated Au.

There have been Pre-hispanic Au-Pt alloy experimental simulations made in different ways. P. Bergsøe [1937], observing semi-finished pieces from Tumaco-La Tolita, deduced the possible technique used: “the small grains of Pt were mixed with a little gold dust and small portions placed upon a piece of wood charcoal”. Gold melts and coats Pt grains; a pasty sintered mass is formed which withstands hammer blows, especially when hot. “By alternately forging and heating it is possible gradually to build up an homogeneous mixture”.



FIGURE 1 MAP FROM THE TUMACO-LA TOLITA REGION IN SOUTH AMERICA

He imitated some alloys using autogenous welder but did not reported either obtained macro or

¹ Presumably sintered after an initial agglomeration with melted Au

microstructures. Bergsoe also proposed a way to platinum plating objects: heating two previously made alloys, one rich in Pt and the other rich in Au then hammered out with a few intervals for annealing. Scott and Bray [1980, 1984] and Scott and Bouchard [1988] worked extensively on the Au-Pt alloys and corroborated the technique envisaged by Bergsoe. He made Au-Pt samples using alluvial Platinum. Scott and Bray [1994] proposes that plating can also be produced by laying Pt particles on the gold, heating to allow diffusion and then finishing with light hammering. Handwerker et al. [1991] prepared samples of 85% Au - 15% Pt by weight with Au and Pt wire and heated up to 1100°C and 1200°C for maximum 7 min. After examining the microstructure and comparing it with a Pre-hispanic object, they concluded that the Pt-Au objects were never heated to 1100°C and, probably, never contained a liquid phase. Bustamante et al. [2006] made several Au-Pt alloys (50% Pt by weight) from alluvial Au and Pt materials, at 1100°C and different times (10 minutes, 2 and 13 hours). Some of them were mechanically worked and annealed at 800°C. They compared their microstructures with some Pre-columbian pieces and agreed with the fact that artisans used the gold melting temperature to sinter, suggesting that they could use ceramic molds as dies to get the desired shape when hammering the pieces. Noguez et al. [2006] simulated experimentally 2 alloys using commercial pure gold and Pt in small pieces: 88% Au - 12% Pt and 30% Au - 70% Pt. The gold was melted around the Pt and then cold and hot hammered continuously with annealing intervals at 950°C, until an homogeneous alloy resulted. G Villegas [2009] in her B.S. thesis, presented several variations in technical experimentations: particle sizes, temperatures, foldings and different platings which made us understand better the contributions to several parameters when simulating Au-Pt alloys. Meeks et al. [2002] based on microstructure and microanalysis observations of a plated Pre-hispanic piece, deduced a technique where two alloys, not particles, were used for plating and then, heating and hammering with some other details to get the final plating piece.

Some of the findings using solid state diffusion by the authors have been reported [Noguez et al., 2009]. The intention of this paper is to show the fabrication of alloy replicas in order to understand the type of processes used by Pre-hispanics and to widen the existing metallurgical models for them. The experience on making some Au-Pt alloys is presented. Solid state

diffusion at 950°C, starting from commercially pure Au and Pt was used to produce three types of alloys: a) "sintered" Au-Pt alloy b) plating Pt over gold alloy and c) Pt - Pt sheet. A special intention in the experimentation was to pay attention to some aspects which influence the diffusion processes in the solid state.

Metallurgical and Other Considerations on Solid State Diffusion for the Au-Pt Replicas

Ancient craftsmen and blacksmiths from all over the world used definitively solid state diffusion processing when working with metals, even if precise details are not known. Archaeologists repeatedly mention that the tradition of hammering (hot and cold forging) and annealing sheets of metal could be one of the preferred techniques in the Pre-hispanic South American metallurgy [Lechtman, 1984]. Au and Pt form substitutional alloys, and both Au and Pt have high melting point, which means they are slow diffusers even at relatively high temperatures. At 950°C the diffusivities are: $D_{Au} = 7.66 \cdot 10^{-11}$ and $D_{Pt} = 1.95 \cdot 10^{-11}$ cm²/s [Noguez et al., 2006]. In spite of that, nowadays solid state diffusion processes are analyzed in different ways that explain the different contributions to solid state diffusion. The processes are named as: diffusion bonding, diffusion alloying, massive diffusion bonding, diffusion in mechanical alloying, hot forging diffusion, etc. Diffusion bonding is defined as a *"process in the solid state for making a monolithic joint through the formation of bonds at an atomic level, as a result of closure of the matting surfaces due to the local plastic deformation at elevated temperature which aids interdiffusion at the surface layers of the materials being joined"* [Shirzadi, 2009]. It is normally conducted in vacuum or a protective atmosphere to avoid the detrimental oxidation; and it is performed at around 50-90% of the melting point. There has been many techniques developed to overcome the limitations imposed by different materials. Diffusion alloying presents a very similar definition: *"a thermally creation of superficial bonds between particles and alloying elements by high temperature diffusion in an annealing furnace"* [Ruas and Guo, 2001]. Mechanical alloying supposes the creation of surfaces through fracture each time the force is applied and even the creation of chemical composition variation at the surface until the homogeneity is reached [Lu, Lai and Zhang, 1997]. Hot forging has been studied in detail theoretically and practically regarding its contribution to the diffusion homogenization. The authors propose that

the enhanced diffusion is due to the recrystallization effects and geometry change, being the recrystallization the major contribution [Chen et al., 2011].

Undoubtedly, a solid state process for making Au-Pt alloys has various diffusive characteristics of the mentioned processes. There is a diffusion bonding initially, without the drawbacks and limitations provoked by the oxide films because both noble metals do not oxidized at any temperature. There is no need for a special preparation of the surface or the use of protective atmosphere. The used temperature is in the range of 50% - 90% of the melting point (or solidus of the alloy) and pressure is applied for some time. When the mechanical work is applied some plastic deformation takes place, flattening the initial sheet formed. Hammering is a compressive force which does not work-harden the gold as other metals², but at the Pt-Au surface it is possible that some work-harden exists, which accelerates locally the diffusion by means of creating defects in the structure. This contributes to lower the energetic barrier to interdiffusion. The hot forging also recrystallizes the sample producing more grain boundaries, another defect source of speeding the diffusion. By folding the formed sheet several times, the contact between different surfaces is forced, therefore the diffusion distance and time diminish. Verhoeven and Clark [1998] have demonstrated that the various foldings of the steel strips when fabricating a Damascus blade by hot forging diminish the diffusion distances for both, C and Mn, which enhances the diffusion, more for the interstitial than for the substitutional atom. Finally, when the sample is annealed in a furnace we could say that a normal interdiffusional process is happening.

From the point of view of processes and theoretical solid state diffusion, there are many aspects which make the fabrication of the Au-Pt alloys be an almost straightforward process without the need of an initial melted gold coating on the Pt particles. There is no need to get to the gold melting temperature.

A homogeneous alloy could be viewed either macroscopically or microscopically or both. The gold and platinum binary phase diagram is presented in Figure 2. It can be seen that the Au-Pt system has a

region of immiscibility. Alloys below 15-18% Pt are monophasic, and all others are biphasic, but it is considered in general a full solubility diagram.

Nevertheless, macroscopically, the alloys are homogeneous. The difference of the low and high Pt alloy will be the colour: a golden colour for the former and a gray silver one for the latter. For what it is known from the Pre-hispanic cultures, colour was fundamental (not properties, as today). Gold colour resembles the sun, silver colour, the moon. Pre-hispanic Andean people called gold the sweat of the sun and silver, the tears of the moon. Both, sun and moon, dictated the cycles of life and were worshiped in those times. We can infer that the making of the range of alloys from gold to silvery colour was related to their cosmic religious view. It is undoubtedly that when they made the alloys, it was the superficial (macroscopic) colour what was important, as well as the shape and size of what they intended to show.

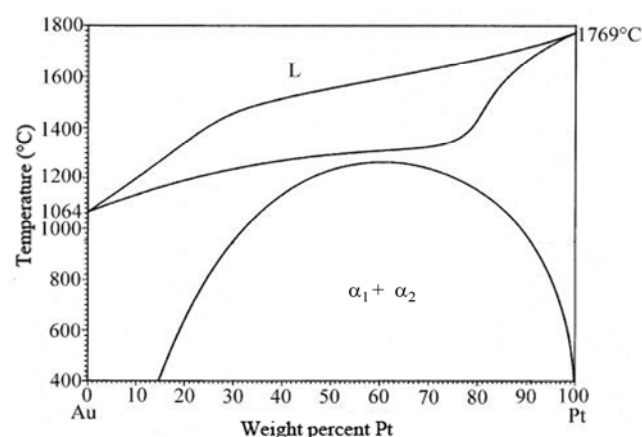


FIGURE 2. THE AU-PT PHASE DIAGRAM SHOWING THE IMMISCIBILITY AREA

As for the platinum plated alloys, all the authors mentioned in the introduction, agreed in proposing a solid state process varying in the specific technique.

One of the main objectives in this work is to fabricate Au-Pt alloys by the all solid state process: hot and cold forging plus some time in the furnace. The sequence in getting the full homogenized alloy will be compared to the one of another alloy fabricated with the initial liquid gold coating and reported in [Noguez et al., 2006]. The time to get a uniform macroscopic colour is recorded. The intention is to get the homogeneity at the microscope and with microanalysis.

Regarding the platinum plated process, the objective is to fabricate a plating alloy and to visualize the process from the diffusion point of view.

² Gold is the most malleable and ductile material. Ramsey [1973] sets that pure gold can recrystallize at room temperature and Tawara, Matsukawa and Kiritani [2003] states that there could be a dislocation-free plastic deformation in gold. These findings could explain the special characteristics of this element.

Experimentation

In order to get the objectives, three types of alloys will be fabricated:

1. Au 82% - Pt 18%. Composition close to two Pre-hispanic objects [Bergsoe, 1937; Handwerker et al., 1991] by solid state processing.
2. Pt-Pt (and Au-Au) by diffusion bonding.
3. Pt plated Au alloy by diffusion bonding.

Parameters and Modus Operandi Selection:

The parameters are selected according to the last experience reported [Noguez et al., 2006]:

Temperature: 950°C. It is 86% below the solidus for the 12% Pt alloy.

Raw materials: Commercially pure Au and Pt.

Particle sizes: 1 x 2 x 0.5mm, simulating sand sizes from the alluvial Pt and Au. Small sheets were used for plating.

Blows: hammer with a woman arm and a steel hammer. The intensity of the loads was measured in a load cell. Because different persons carry on the experiments, there were different loads used indistinctively: high intensity (H) approximate 530 N, medium intensity (M) 480 N, and low intensity (L) 450 N.

Weight of the samples: 1g.

Analyzing modes: Optical microscope, Scanning Electron Microscope (JEOL JSM-5900 LV) with X ray microanalysis.

Procedure

Two alloys type 1 were made, the procedure can be described as follows: a) initially, particles were heated at 950°C for 3 h in a furnace; b) hot forging (900-950°C) with a small torch, folding the sample as needed, measuring the number of blows; c) letting stand for 10 h in a furnace at 950°C; d) analyzing the sample with the microscope and microanalysis. After the first cycle the sequence is: 10 h diffusion at 950°C - hot forging - 10 h diffusion at 950°C -analysis, until getting the homogeneity by microanalysis.

For alloys type 2, the procedure was hot forging with the torch at 900-950°C until getting a small sheet.

For alloys type 3 using a type 1 alloy sheet, Pt particles were placed behind it, heat at 950°C, hot forge, heat in

furnace for 4 h and analyze with microscope and microanalysis.

Results

Type 1 Alloys

As an example, the following figures (3-7) will exhibit several steps in the fabrication of alloy 1. Metallographic samples were prepared as usual and etched with aqua regia (60% HCl and 40% HNO₃).

Figure 3 shows the employed particle size, Figures 4 and 5 give a macro-view and a micro-view of the alloy at 28 h, respectively. A Pt particle is very noticeable in Figure 5.

The variation of concentrations for alloy 1 during the cycles is displayed in Figure 8, as an example of the experimentation, while Table 1 shows the features of each cycle in it. Data are cumulative.

In the course of experimentation, two different persons made the alloys, having different load on her arm, as it was mentioned in the experimentation section. So, final times were different. Alloy 1 was made in 88 h (macroscopically homogeneous at 48 h) and the other one in 40 h (macroscopically homogeneous at 30 h).

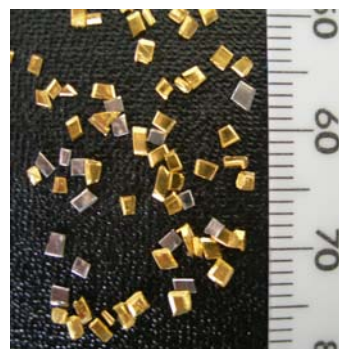


FIGURE 3. PT AND AU INITIAL PARTICLES (SCALE IN MM)



FIGURE 4. MACROSCOPIC APPEARANCE OF ALLOY 1 AFTER 28 H

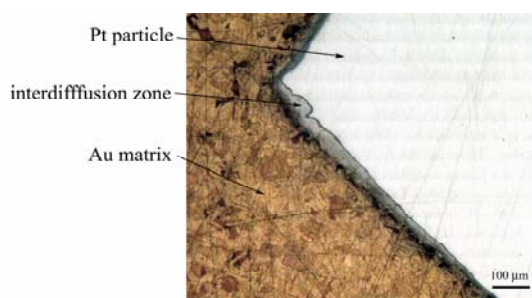


FIGURE 5. MICROSTRUCTURE OF THE ALLOY 1, AFTER 28 H, SHOWING WHITE PT PARTICLES (RIGHT)

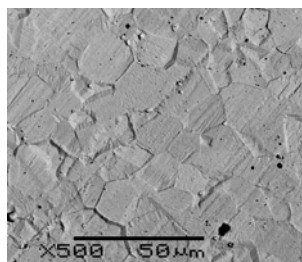


FIGURE 6. AFTER 88 H TOTAL HOMOGENEITY IS OBSERVED. TWININGS ARE VISIBLE

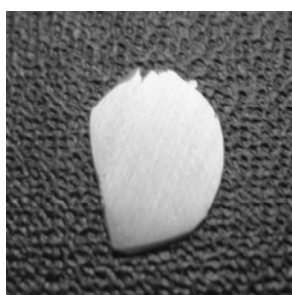


FIGURE 7. FINAL APPEARANCE SHOWING TOTAL HOMOGENEITY

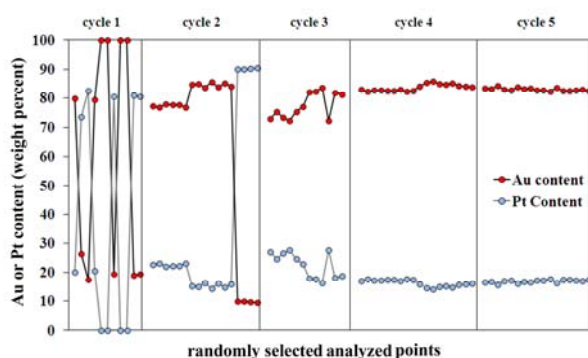


FIGURE 8. ALLOY 1 AU AND PT CONTENT OVER ANALYTICAL POINTS FOR EACH CYCLE. DIFFUSION TEMPERATURE WAS 950°C, WHILE BLOWING-FOLDING TEMPERATURE WAS 730-100°C. FEATURES OF EACH CYCLE ARE SHOWN IN TABLE I

The time to get a visual or macro view of the alloy is roughly half the time to get the homogeneity measured by microanalysis. In Pre-hispanic times, the making of the alloys surely stopped when getting the desired color in the surface; the times vary also depending on the person, so it is possible that time to

make an alloy by solid state is not as long as it could be though.

TABLE 1. CHARACTERISTICS OF DIFFUSION-BLOWING-FOLDING CYCLES FOR ALLOY 1

| Cycle | Diffusion Time (h) | Blows | Foldings |
|-------|--------------------|-------|----------|
| 1 | 8 | 132 | 8 |
| 2 | 28 | 310 | 10 |
| 3 | 48 | 202 | 20 |
| 4 | 68 | 386 | 28 |
| 5 | 88 | 472 | 33 |

Type 2 Alloys

Joining Au particles to Au particles by hot forging is an easy task. It takes around 2-5 minutes and around 20 blows to obtain a sheet using the small particles. Joining Pt particles takes longer: 20-25 minutes with 80 blows. The following figures display the initial and final images.



FIGURE 9. INITIAL AU PARTICLES (SCALE IN CM)



FIGURE 10. INITIAL PT PARTICLES (SCALE IN MM)



FIGURE 11. FINAL AU AND PT PARTICLES (SCALE IN CM)

Type 3 Alloys

A platinum plated sample was obtained using the small 82% Au - 18% Pt alloy sheet fabricated as type 1 alloy and Pt particles.

Scott and Bray [1994] set that Pt particles should cover the alloy before the forging. It was considered better to get the Pt particles behind the alloy, then hot forge and reheat at 950°C in a furnace. The particles were behind for an easy and better hammering, otherwise the particles could jump out. This alloy was made to witness the feasibility of the solid diffusion when plating the Au-Pt. Figures 12, 13, and 14 illustrate different steps during the experimentation.

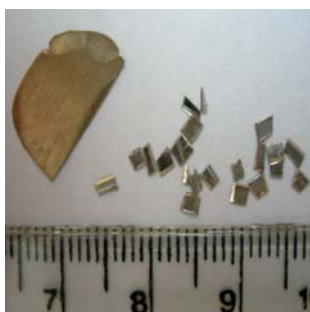


FIGURE 12. INITIAL BASE ALLOY AND PT PARTICLES (SCALE IN CM)

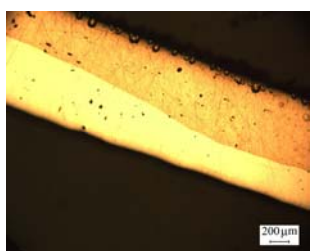


FIGURE 13. LOW MAGNIFICATION STRUCTURE

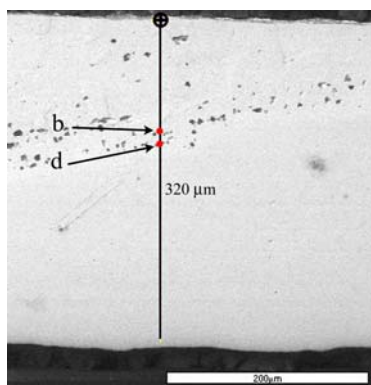


FIGURE 14. SEM MICROSTRUCTURE SHOWING THE LINE WHERE ANALYSES WERE PERFORMED AFTER 4 H DIFFUSION

From Fig 13 and 14, it is clearly seen that the Pt coat is very irregular which could obey to the irregular position of Pt particles and/or to the width irregularities of the initial Au-Pt alloy. Typical solid state diffusion voids are seen in the Gold.

The solid state diffusion occurring in the sample is illustrated in figure 15. The graph plots Pt or Au concentration vs. distance in the transversal direction for a quick comparison between forging and forging plus 4 h diffusion. The composition differences after forging are the squares and circles; and after diffusion are triangles and rhomboids. One can notice that some diffusion went on because Gold and Platinum have interdiffused. There is a difference in composition due to the immiscibility region in the phase diagram -the equilibrium between α_1 low Pt solution, and α_2 high Pt solution-. This difference should have been represented for one straight and vertical line at a very definite distance, with definite compositions; instead there is a slope and it seems there is a range in both, distance and compositions, because of a lack of analytical points.

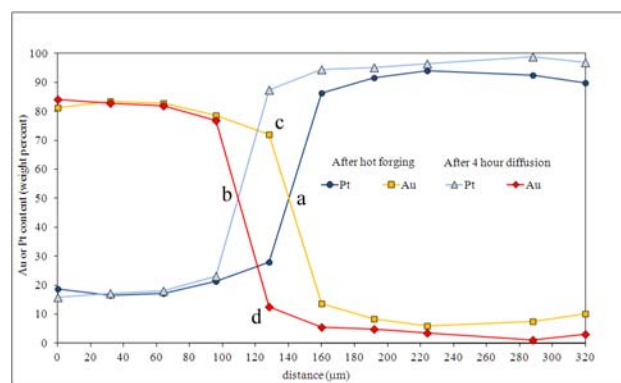


FIG 15. VARIATION IN CONCENTRATION ACROSS THE PLATED SAMPLE AFTER FORGING AND AFTER 4 HRS DIFFUSION. A, B AND C POINTS ARE MARKED IN FIGURE 14

However, after forging, this equilibrium is represented in figure 15 at an average distance of 140 μm (point "a") and, after 4 hrs diffusion, around 110 μm (point b). From another point of view, the point "c", at 120 μm (72% Au and 28% Pt) after forging changes to point "d" (12% Au and 88% Pt) after diffusion. Points "b" and "d" are also indicated in figure 14.

Discussion

With very simple laboratory experiments, it has been demonstrated that Au and Pt particles can be worked by hammering and heating below the melting point of Au, all solid state, to obtain several Au-Pt alloys.

Pt and Au are the most noble metals, no oxides form at any temperature, and they are workable, specially Au-low Pt level alloy.

Because of the former experience reported fabricating Au-Pt alloys using the P. Bergsoe technique [1937]: melting the Au particles around Pt to agglomerate

them, a comparison of the processes is made in Table 2. The theoretical time calculated with one of the engineering models (sinusoidal) is exhibit to visualize the enhancing diffusion during the experimental processes.

TABLE 2. COMPARISON OF EXPERIMENTAL RESULTS WITH A THEORETICAL MODEL

| Alloy Process | Diffusion Time (h) | Blows | Foldings | Time (%) |
|-------------------|----------------------|----------------------|----------|----------|
| Theoretical Model | 548 | | | 100 |
| Au 88% - Pt 12% | 2 in liquid Au + 190 | 240 (L) ^A | 20 | 35.7 |
| Au 82% - Pt 18% | 88 | 476 (M) ^A | 33 | 16.0 |
| Au 82% - Pt 18% | 40 | 630 (H) ^B | 20 | 7.3 |

^A Hot and Cold Forging; ^B Hot Forging; (H) 530 N, (M) 480 N, (L) 450 N

The theoretical model has the higher time, because it do not consider the mechanisms that ease the diffusion by enhancing it during the forging: defects creation, recrystallization, geometry changes, etc. It would be interesting to introduce all this factors in developing a better model. The sinusoidal model could consider just the foldings to shorten some diffusion distances. Experiment 1, from reference [Villegas, 2009] having 2 h diffusion at the Au melting temperature has the largest time in experimentation. Diffusion at melting temperatures does not bring a considerable diffusion, probably because the time is too short, and the enhancement in diffusivity for Au in liquid state from 10⁻¹¹ to 10⁻⁵ is not enough for the time employed. Besides. the forging was done with low force, employing cold and hot forge. It was the first made sample.

Type 1 alloys (1 and 2) from this paper, differ in the number and intensity of blows, besides the hot forging was used exclusively in alloy 2. Both presented recrystallization structure noticeable because of the exhibited twins (see Figure 6).

The considerable reduction in time from the theoretical model sets the feasibility of working all solid state without the need to get the Au melting temperature.

Type 2 alloys diffusion bonding Au-Au and Pt-Pt particles are the easiest to fabricate in solid state especially because they are noble metals.

Type 3 alloys Platinum plated over Au-Pt alloy, with hot forging and following diffusion shows the advancement of diffusion as interdiffusion when they are heated in the furnace after forging.

A model for fabricating of alloys all solid state diffusion, without reaching the Au melting temperature, is proposed and validated. This is different to what the authors in the literature have assumed. Regarding the cladding, it is proposed and proved by a different technique to obtain it.

Conclusions

It is technically possible to fabricate Au-Pt alloys without using the Gold melting temperature. Consequently, Pre-hispanic inhabitants could use this process when making their alloys and objects, reaching a pertinent engineering solution. From this, another model in archaeometallurgy for Au-Pt alloys in Pre-hispanic times is proposed.

Acknowledges

The authors give special thanks to Guadalupe Villegas for working on the topic and to Ivan Puente and Guillermina González for their work with SEM, including Microanalysis. Also to the B.S. student Luz Elena Rosales for their disposition to support with laboratory work when needed.

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María Eugenia Noguez. Born in Mexico City, Mexico on June 4th 1947; B.S. in Metallurgical Chemical Engineering at Facultad de Química, Universidad Nacional Autónoma de México (UNAM, 1964-1968). M.S. in Metallurgical Engineering at School of Engineering, University of Pittsburgh, 1973. Major field of interest: physical metallurgy and engineering education.

She started to work professionally in the Automotive Industry as a Quality Laboratory Technician and in the Foundry Industry as a Manufacturing Engineer. At UNAM, she started as a Lecturer in 1968. After graduate studies, she became an Associate Professor in 1974 and Professor in 1984. Actual position: Professor at the Facultad de Química, UNAM. She has directed 37 B.S. theses. She has published around 80 papers in different areas: solidification, structure of materials, archaeometallurgy and engineering education. She has presented research and educational works in 85 metallurgical congresses.

Ms. Noguez obtained the Latin American Program of American Universities (LASPAU) scholarship to do graduate studies in USA (1971-1973). At UNAM Facultad de Química, she was elected member of the Technical Council as a student (1965-1968), then as a teacher for 3 periods (1976-1983, 1983-1989, 1995-2001); coordinator of the BS studies on Metallurgical Chemical Engineering (1979-1981), and coordinator of graduate studies in Metallurgy (1983-1985); she won the "Ernesto Rios del Castillo" award lecturer position (1995-1997). She has been member of the AFS, The Metallurgical Society of AIME, AITS and TMS.



Guillermo Salas. Born in México City, México on November 20th, 1950; B.S. in Metallurgical Chemical Engineering at Facultad de Química, Universidad Nacional Autónoma de México (UNAM, 1969-1973). Foundry Specialty at Facultad de Química, UNAM (1976). His major field of interest is students

learning.

He worked professionally partial time in the industry of manufacture for near 6 years. At the National University of Mexico, he started as a lecturer in 1974. He became an Associate Professor in 1979 and Professor in 1987. Actual position: Professor at the Facultad de Química, UNAM. He has directed 30 B.S. theses, published more than 80 papers in different areas: solidification, structure and properties of materials, archaeometallurgy and engineering education, and presented research and educational works in 85 metallurgical congresses.

He obtained his scholarship to graduate studies from the American States Organization (AEO). At the University of Mexico, School of Chemistry, He was member of the Technical Council as a teacher for the period 1989-1995;

chairman of the Department of Metallurgical Chemical Engineering (1979-1981). He has been member of the AFS, The Metallurgical Society of AIME, and TMS.



José Ramírez. Born in México City on May 23th, 1963. He obtained the B.S. in Metallurgical Chemical Engineering in 1992 at the Facultad de Química of the Universidad Nacional Autónoma de México (UNAM), where he also completed graduate studies in

Mechanical Engineering at Facultad de Ingeniería. His major fields of studies are physical metallurgy.

He was Lecturer (1993-1994) and has since been Research Assistant. In addition, he currently serves as Adjunct Professor in the Facultad de Química, UNAM. He has collaborated on several research projects, in presentation of papers at around 50 congresses and in the publication of 20 articles on the topics of his interest: physical metallurgy, archaeometallurgy and engineering education.

Mr. Ramírez has been member of ASM, TMS and the Mexican Chemical Society.